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Dynamics of optical excitations in a ladder-type π-conjugated polymer containing aggregate states

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The dynamics of optically generated excitations in a poly-(para)-phenylene–type ladder polymer has been investigated by means of femtosecond pump-probe as well as femtosecond luminescence up-conversion spectroscopy. In addition to the bulk emission a low-energy emission band assigned to physical aggregates is observed. By comparing the photoluminescence (PL) decay of the bulk emission with the time-resolved PL traces of the aggregate emission we show that in the presence of aggregates the dynamics of the initially generated excitons are determined by transfer from bulk to aggregate states. Additionally stimulated emission (SE) as well as photoinduced absorption are observed for different spectral regions in the pump-probe experiments. The SE is ascribed to bulk singlet excitons as evidenced by the correspondence of the temporal behavior of SE and the decay of bulk emission, whereas the PA is attributed to spatially separated excitons. [S0163-1829(96)06027-4]

I. INTRODUCTION

The potential application of conjugated polymers in optoelectronic devices has stimulated intense research activities concerning the yield and the dynamics of elementary excitations in this class of materials.[1,2] The nature of the photogenerated elementary excitations and their relaxation dynamics are of both theoretical and practical importance. Theoretical work, in general, focuses on the optical properties of ideal, fully elongated single polymer chains yet neglects possible interchain interaction leading to the formation of aggregate states.[3,4]

The material investigated is a poly-(para)-phenylene–type ladder polymer (LPPP).[5] It is soluble and structurally regular with an average molecular weight of up to 12 000 corresponding to 30 monomer units. The characteristic optical properties of LPPP originate from the stabilization of the backbone chain against torsional displacement of the phenyl ring by covalent bridging. This leads to a negligible Stokes shift due to structural relaxation as compared to systems in which coupling of the excited states to torsional modes of the chain is important.[6–8]

So far, the optical properties of LPPP have been investigated mainly by steady-state photoluminescence (PL) as well as picosecond (ps) streak-camera PL measurements. From these experiments information was obtained concerning the dynamics of the optically generated excitations and the existence of aggregate states acting as traps.[9]

In the present paper, we focus on the excitation dynamics of solid LPPP in the excited state by means of femtosecond upconversion and femtosecond-pump-probe spectroscopy. In particular, we show that the capture of elementary excitations (singlet excitons) by aggregate states dominates the observed fast PL decay. In addition, the pump-probe technique provides significant information about the existence and the dynamics of excitations other than singlet excitons that manifest themselves by a photoinduced absorption (PA). Recent studies on poly(phenylene vinylene) (PPV) have shown that there is a competition between stimulated emission and photoinduced absorption, the latter being attributed to Coulombically bound electron-hole pairs located on different chains and/or segments.[10] The present results demonstrate that the simultaneous occurrence of stimulated emission and PA due to Coulombically bound electron-hole pairs on adjacent segments (spatially separated excitons) is a general phenomenon in conjugated polymers. Time-dependent pump-probe experiments allow one to monitor the complex decay pattern of both neutral and charged excitations.

II. EXPERIMENT

All experiments were performed on LPPP synthesized via a two-step precursor route bridging properly substituted precursors.[11] The samples were either bulk films or polymer blends of LPPP and polycarbonate (PC) in a 1:9 ratio by weight, both spin coated onto quartz substrates. The chemical structure is shown in the inset of Fig. 1.

The femtosecond luminescence experiments employing the up-conversion technique were performed on bulk films of LPPP. For reasons of optimizing the optical density polymer blend films were used for the pump-probe experiments. We wish to point out that dilution of the LPPP does not affect its spectroscopic properties as evidenced by the similarity of the PL spectra. The experiments that delineate the dynamics of

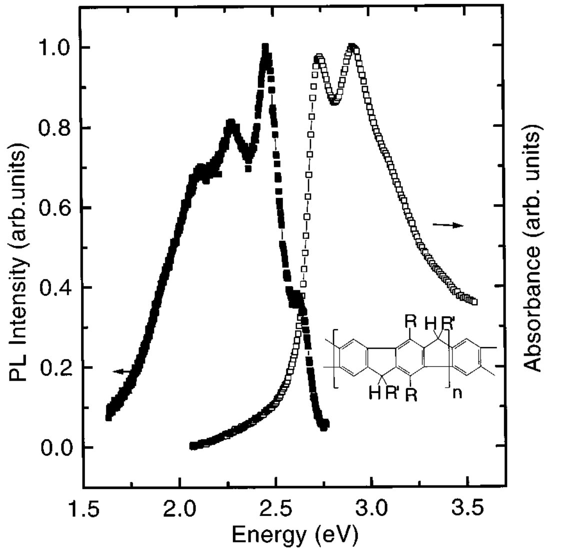

FIG. 1. Absorption and photoluminescence spectra of LPPP. The PL is excited at 3.1 eV. The inset displays the chemical structure of the LPPP, *R* and *R'* are *n*-hexyl and 1.4-decylphenyl, respectively.

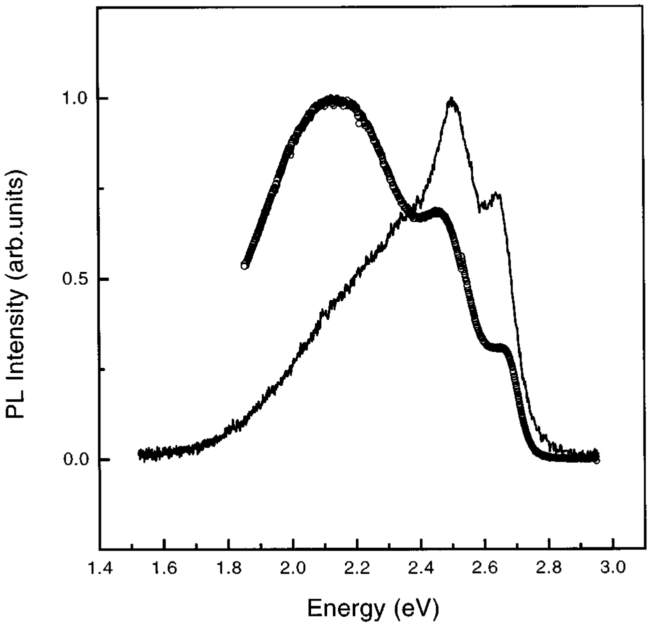

FIG. 2. Low-temperature time-integrated PL spectra of two different LPPP films. The excitation energy is 3.1 eV.

optical excitations were performed with a Kerr-lens mode-locked Ti:sapphire laser producing pulses at 1.56 eV with 100-fs duration at a repetition rate of 76 MHz. The samples were excited by frequency doubled laser pulses at 3.1 eV, i.e., 0.3 eV above the pure electronic transition. The excitation power was 20 W/cm^2 equivalent to 10 nJ per pulse. The emitted luminescence from the sample was focused dispersion free onto a nonlinear optical crystal of β-bariumborate. Up-converted signals are generated if the luminescence and the gating reference pulse (1.56 eV) overlap temporally within the crystal. The up-converted signal was dispersed in a monochromator and finally detected by a cooled GaAs photomultiplier. The temporal resolution of the setup was about 200 fs while the spectral resolution was about 20 meV. Additionally, femtosecond pump-probe experiments with a spectrally broad white-light continuum as probe light were performed. For primary excitation we used laser pulses of 160-fs duration at an energy of 3.1 eV having pulse intensities of up to 4 μJ with a repetition rate of 1 kHz. There was no indication of either nonlinear effects or photochemical degradation at this excitation dose. The excitation pulses were obtained by frequency doubling one part of the amplified output of a Ti:sapphire laser while the other part was used to generate white-light continuum pulses in a laminar water cell. Differential transmission (DT) spectra were recorded within the spectral range from 1.6 to 2.8 eV. All spectra were corrected for group velocity dispersion. Moreover, we have measured the DT as a function of time delay in a narrow spectral range using a 10-nm broad portion of the white-light continuum as a probe. In this case the probe energy was selected by interference filters.

III. RESULTS

Figure 1 presents the absorption as well as the time-integrated photoluminescence spectra of a LPPP/PC blend. The absorption spectrum consists of the $S_1 \leftarrow S_0$(0-0) transition at 2.74 eV followed by its dominant vibronic satellite at 2.91 eV. All bands are inhomogeneously broadened bearing out a Gaussian width σ of about 0.07 eV. Remarkably, the absorption spectrum exhibits a pronounced low-energy tail extending well below 2.1 eV. The emission spectrum consists of the fundamental electronic $S_1 \rightarrow S_0$(0-0) transition at 2.66 eV followed by vibronic progressions. The low intensity of the $S_1 \rightarrow S_0$(0-0) transition compared to the dominant first vibronic transition is caused by reabsorption. It is important to mention that the emission spectrum is a superposition of the bulk $S_1 \rightarrow S_0$ spectrum and a broad featureless low-energy band centered around 2.1 eV as has been shown in greater detail in previous work.[9] Figure 2 shows the cw-PL spectra of two bulk LPPP films containing different amounts of aggregates upon excitation at 3.1 eV. The spectra resemble those observed in the blend system except for the intensity of the broad yellow emission band. The latter depends on the details of the film preparation resulting in the incorporation of different amounts of aggregates.[9]

Figure 3 displays luminescence transients detected at different spectral positions after excitation with photon energies well above the optical gap (3.1 eV). The luminescence rise follows the response function of the experimental setup whereas the observed decay becomes slower as the spectral detection window is shifted towards lower energies. All decay functions are nonexponential, reflecting the occurrence of nonradiative decay channels.[12] Probing the PL decay at photon energies corresponding to the structureless emission a totally different transient behavior is observed. In that case the luminescence rise is not determined by the response of the experimental setup. Here we observe a finite PL rise completed after approximately 8 ps as shown in Fig. 4. It has to be attributed to a filling process from energetically higher lying states into those responsible for the structureless emission centered around 2.1 eV. Further insight into this scenario comes from the time-resolved luminescence spectra

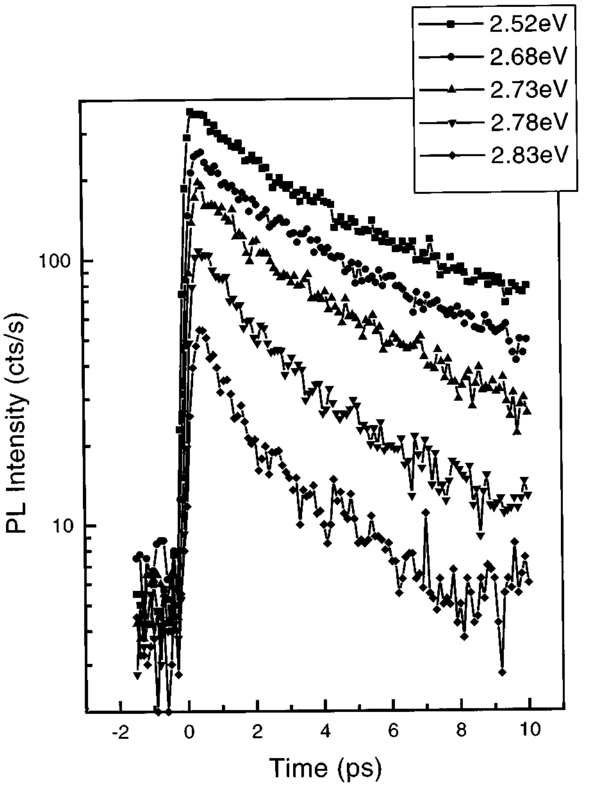

FIG. 3. PL transients monitored at different spectral positions. The excitation energy is 3.1 eV.

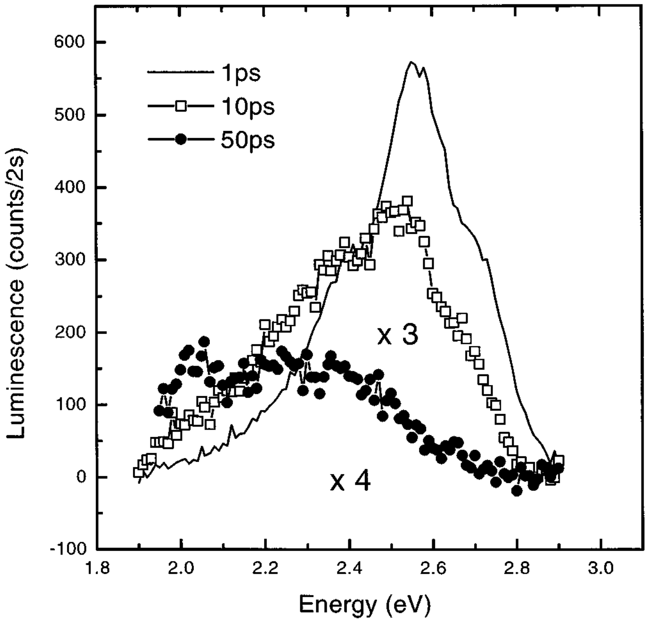

FIG. 5. Time-resolved spectra of LPPP for various delay times. The spectra taken after 10 and 50 ps are scaled with factors of 3 and 4, respectively.

presented in Fig. 5. Within 1 ps the spectrum perfectly maps the cw spectrum of pure LPPP in solution.[13] The purely electronic transition associated with the shoulder at ≃2.7 eV is followed by a vibronic progression at lower energy. 10 ps after excitation a transient redshift due to energetic relaxation known from other polymer systems[14] is observed. At longer times a broad yellow emission evolves at the expense of the bulk emission and dominates the spectrum at longer time delays as shown in the time-resolved spectrum monitored 50 ps after excitation.

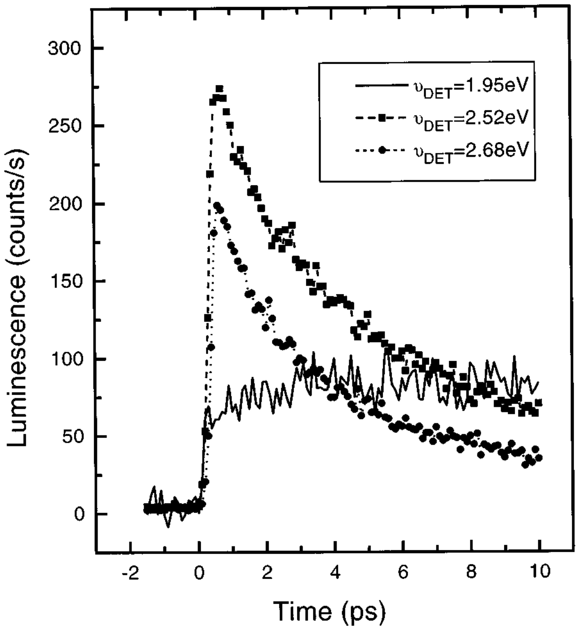

FIG. 4. PL transients detected at photon energies within the bulk emission and at the emission of the aggregate states (1.95 eV).

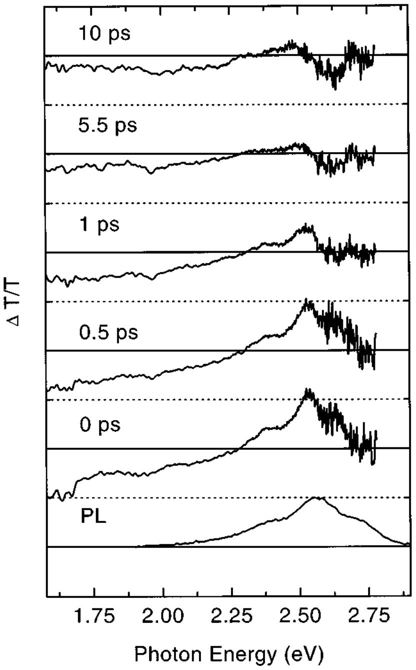

FIG. 6. Differential transmission (DT) spectra for excitation at 3.1 eV for various delay times between pump and probe pulse. The dotted lines represent a change in probe transmission of 10%. For comparison the normalized PL spectrum of bulk excitons is plotted at the bottom.

Figure 6 depicts DT spectra after excitation at 3.1 eV for various delay times between pump and probe pulses. The cw PL spectrum is plotted for comparison. At zero time delay

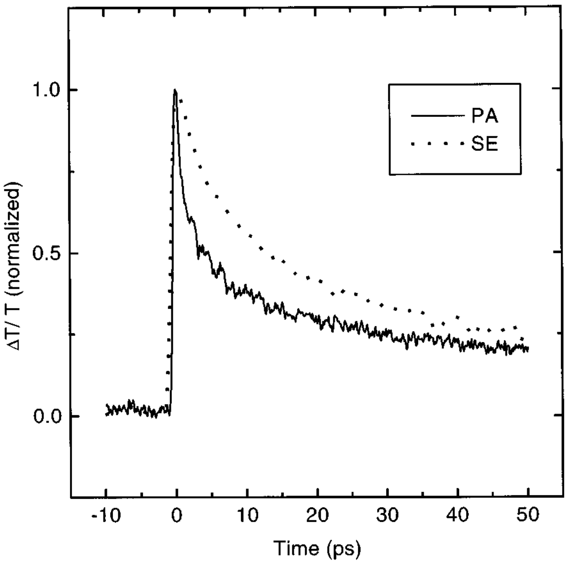

FIG. 7. Temporal behavior of the stimulated emission (SE) at 2.48 eV (dotted line) and the photoinduced absorption (PA) at 1.91 eV (solid line).

the spectrum is characterized by two main features. In the spectral range from 2.38 to 2.58 eV positive differential transmission with $\Delta T/T$ up to 10% is observed. The shape of the positive DT spectrum resembles the steady-state PL spectrum but bears out a bathochromic shift of about 70 meV upon increasing the delay time from 0 to 10 ps. At lower photon energies the spectrum is dominated by a broad and structureless negative DT signal ranging from 1.37 to 2.29 eV, indicating a photoinduced absorption. With increasing delay time there is a redistribution of oscillator strength within the DT spectra because the positive DT signal decays more rapidly than the negative DT signal. Finally the positive DT in the spectral region of the 0-0 transition is overcompensated by the PA and becomes negative. Normalized transients of both the positive DT signal (labeled SE in Fig. 7) as well as of the PA are shown in Fig. 7. Within the time resolution of the experimental setup both transients rise instantaneously and exhibit a nonexponential decay.

IV. DISCUSSION

By comparing the absorption and emission spectra we assign the yellow emission centered around 2.1 eV to aggregate states formed by subunits of different polymer chains in closely packed solid LPPP films. The yellow emission stems from species that exist in the ground state as demonstrated by the finite absorption found in the corresponding spectral region.[9] Since chemical impurities can safely be excluded and since site-selective fluorescence measurements indicate a concentration dependence of the intensity of the yellow emission band, it is plausible to associate the traps with preexisting aggregates in which interchain interaction is enhanced. Importantly, the intensity of the aggregate band is much stronger in emission than in absorption. This is an unambiguous signature of efficient energy transport to those states.

Information concerning the relaxation of excitations taking place after their generation is deduced from luminescence transients monitored at different spectral positions. The variation of the characteristic decay time upon lowering the detection energy, the accompanied transient redshift of the emission spectrum, and the nonexponential decay pattern are characteristic features of energetic relaxation of neutral excitations within an inhomogeneously broadened density of states (DOS).[15] The PL spectrum taken 1 ps after excitation contains two important messages: (i) The vibronic excess energy of ≈0.3 eV is dissipated on a sub-200-fs time scale. This is in accord with previous results for PPV.[14] (ii) Except for the reduction of the $S_1 \rightarrow S_0$(0-0) transition caused by reabsorption it is the mirror image of the absorption spectrum shown in Fig. 1. It is particularly noteworthy that the center of the deconvoluted 0-0 band is redshifted for longer times. This clearly indicates that the entire Stokes shift of the cw-PL spectrum is due to electronic spectral relaxation rather than to molecular relaxation. At the beginning of its random walk an excitation will execute nearest-neighbor jumps only. If the detection window is located at the high-energy side of the DOS the population decay is determined by fast hopping to acceptor states at lower energies. The strong dependence of the decay time on energy reflects the energy dependence of the hopping rate, which gradually slows down as the excitations relax towards the tail states of the DOS. Consequently, the overall decay pattern of donor states in a certain spectrum window within the DOS is controlled by both depopulation towards lower-energy states and refilling from higher-lying states. Taking into account that the latter process is the fastest one, one should, in general, observe transients featuring a finite rise followed by a slower decay. The rise should become more pronounced upon shifting the detection window towards lower energies because the probability to find a site lower in energy next to a donor site decreases. This scenario is reflected by the transients monitored within the spectral range of the bulk emission in Fig. 3. Due to their lower-lying energy level fluorescent traps, e.g., aggregates, are prominent candidates for observing transients featuring accumulation effects. The transient luminescence monitored at 1.95 eV exactly exhibits the behavior discussed above as shown in Fig. 4. Remarkably, within the first 8 ps the decay of the bulk emission exhibits exponential behavior and gradually becomes nonexponential for longer times. Within the time range of the exponential behavior the decay bears out a time constant of 8 ps. This concurs with the time constant of the filling process observed at the spectral position of the aggregate emission as depicted in Fig. 4. Consequently, we conclude that the population of the aggregate states results from highly efficient energy transfer of excitations from the bulk states. Observing only a short rise component of the luminescence signal monitored at detection energies in the tail states of the DOS indicates, however, that only a fraction of excitations accumulate in the bottom states, the remainder being captured by nonradiative traps in analogy to what has been observed with PPV.[14] The $1/e$ decay time of the subsequent PL decay increases by more than an order of magnitude upon shifting the detection window from the spectral range of the bulk emission into the range of the aggregate emission.[9] This is in accord with the decreasing number of available states at lower energy. However, comparison of measurements of the quantum efficiency of solid LPPP (Ref. 9) with LPPP in solution (Ref. 7) indi-

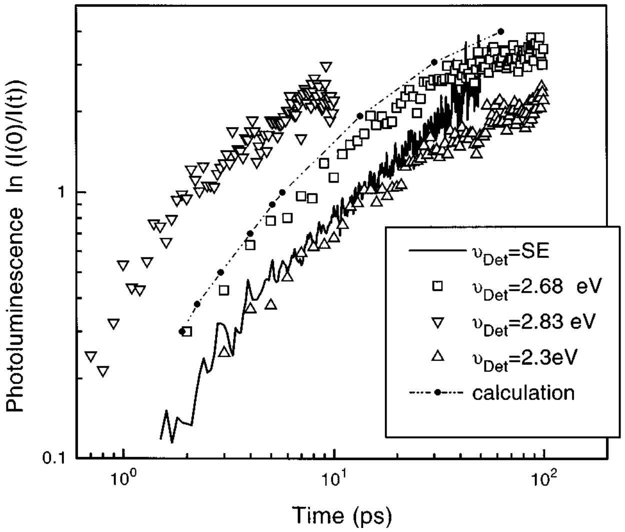

FIG. 8. Smoothed decay curves of the PL in LPPP replotted on a double-logarithmic $\ln(I_0/I)$ vs time scale. The dashed line is a model calculation taken from Ref. 17. SE monitored at 2.48 eV is plotted for comparison.

cates that nonradiative decay channels must also be operative in solid LPPP and compete with the transfer of bulk excitations into luminescent aggregate states.

The time dependence of incoherent energy transfer in amorphous materials is often analyzed in terms of a Kohlrausch-Williams-Watt (KWW) type stretched exponential[16]

$$I(t)=I_0\exp[-(t/t_0)^{\beta}].$$

Both independent single step processes and sequential multistep processes can lead to such a behavior provided the system is completely random. To check whether or not the present data are in accord with this formalism it is useful to plot $\ln I_0/I(t)$ versus t on a double-logarithmic scale as shown in Fig. 8. In such a plot an exponential decay process leads to a straight line with slope 1. For short times the slope of the resulting plots is in fact close to unity, indicating that the fastest decay process occurs with a well-defined rate. This is in accord with the notion that at the beginning of its random walk an excitation only executes nearest-neighbor jumps. Upon probing at 2.83 eV, i.e., above the center of the $S_1 \leftarrow S_0$(0-0) absorption band the initial exponential portion of the PL transient decays with a $1/e$ decay time of ≈ 1.8 ps and tails off in a KWW-like fashion with an exponent $\beta \approx 0.4$ in analogy to what has been reported earlier for PPV. It is straightforward to associate the fast decay with nearest-neighbor hopping within the distribution of bulk states. On shifting the spectral observation window towards lower energies the entire decay pattern is shifted along the time axis without losing any of its basic features. At a detection energy of 2.68 eV, i.e., 0.06 eV below the $S_1 \leftarrow S_0$(0-0) maximum the initial $1/e$ decay time has increased to 7 ps. This is basically the initial rise time of the defect emission indicating that within this time-energy window it is mainly energy transfer from bulk to aggregate states that controls the excitation dynamics. For longer times the slope of the $\ln I_0/I(t)$ versus time curve in log-log representation deviates from unity equivalent to energy migration becoming dispersive reflecting the distribution of jump rates. This is due to the dilution of suitable nearest-neighbor acceptor sites once an ensemble of excitations has relaxed towards the tail of the distribution of states and the growing importance of non-nearest-neighbor jumps. In the long time limit values $\beta \approx 0.4$ and 0.25 are obtained after probing at 2.83 and 2.68 eV, respectively. The latter value is slightly less than normally found for conjugated polymers.[17]. This is another signature of relaxation into aggregate states. The distribution of those states is superimposed onto the distribution of bulk states, implying that the convoluted density of states tails off much slower than an unperturbed Gaussian.

Spectral relaxation and PL decay in conjugated polymers have been studied before via simulation assuming that excitation transport occurs via hopping among different segments of the polymer chain(s). These segments were considered as sites forming a random 1D chain with constant intersite separation featuring a Gaussian distribution of excitation energies and coupled via dipole-dipole interaction (Förster coupling). It has further been assumed that 10% of the sites are traps. In Fig. 8 the result of the simulation for the depopulation of bulk states located at an energy σ above the center of the $S_1 \leftarrow S_0$(0-0) band, whose inhomogeneous broadening is characterized by a Gaussian width σ, is included. The similarity with the present experimental results is striking. It demonstrates that the excitation dynamics in LPPP can be interpreted consistently in terms of spectral relaxation within a density of states that carries a long tail due to the presence of aggregate states.

The time dependence of the transients detected via pump-probe experiments (Fig. 7) is similar to those shown by the PL transients in Fig. 8. Within the first 8 ps the positive DT decay is monoexponential. Extrapolating the initial linear portion of the double-logarithmic $\ln I_0/I(t)$ versus time plot to an ordinate value $\ln I_0/I$ $(t=t_{1/e})=1$ yields a decay time of $t_{1/e}=8$ ps. For longer times it gradually becomes nonexponential. The decay constant as well as the spectral position and the shape of the positive DT concur with the data concerning the bulk emission inferred from the up-conversion fluorescence measurements. It is particularly noteworthy that the decay function fits perfectly into the PL decay pattern probed at variable detection energies (Fig. 8). This not only proves that positive DT and PL originate from the same species, i.e., bulk singlet excitons prior to trapping, but also testifies to the absence of additional decay channels, such as bimolecular exciton fusion that might be considered to occur at the photon dose employed in the pump-probe experiment. The decay of both the DT and the PL signal reflects the decay of the initial singlet exciton population due to both spectral relaxation and trapping by structural defects as already discussed above. The fact that the positive DT signal appears to decay faster again at long times compared to the PL is probably an artifact caused by the superposition of the longer living (negative) photoinduced absorption signal. Following previous work on PPV it is straightforward to assign the positive DT signal to stimulated emission (SE) from bulk singlet states. It is noteworthy that no stimulated emission from longer lived defect states is observed. The likely reason is their smaller radiative yield.

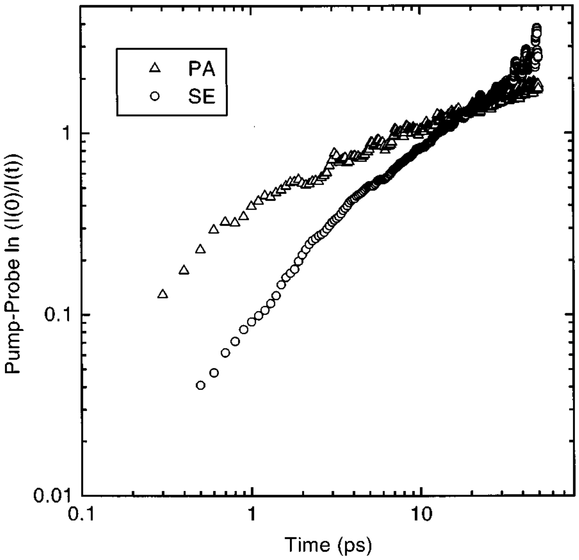

FIG. 9. Double-logarithmic plot of $\ln(I_0/I)$ vs time for the stimulated emission (○) and the photoinduced absorption (△).

The PA signal probed at 1.91 eV where no SE is observed exhibits a different time dependence (see Fig. 9). The decay is highly nonexponential bearing out a KWW exponent close to 1/3. Only for $t \leqslant 1$ ps can an exponentially decaying component with a $1/e$ decay time of about 2 ps be distinguished. The different decay pattern of SE and PA indicates that both must originate from different species. From these data we can rule out bulk singlet excitons as the origin for the PA in contrast to previous conclusions by Leng *et al.*[18] for PPV. Taking into account that the rise of the PA signal is instantaneous one can safely exclude triplet excitons as the fundamental excitations for the PA signal as well since those should be formed on a nanosecond time scale. Aggregates cannot be responsible for the PA signal either since no time lag is observed. The only remaining alternative candidates are spatially separated excitons where the electron and the hole are Coulombically bound yet located on different chains. They have been invoked before in PPV.[19] Consistent with this assignment is the almost featureless spectrum extending from $\simeq$2.2 eV towards lower energies, which is suggestive of transient absorption by single charged species, spectrally blurred by the presence of the respective counter charge at random distance.

Finally, we comment on the formation process for spatially separated excitons. The average binding energy of a spatially separated exciton cannot be less than that of an on-chain singlet exciton otherwise no intrinsic bulk fluorescence should occur. Taking into account that the photon energy of the exciting pump pulse exceeds the $S_1 \leftarrow S_0$(0-0) approximately 0.35 eV, which is on the order of a typical exciton binding energy in conjugated polymers,[20] it appears plausible that spatially separated excitons are generated with finite probability from a hot singlet state and at a rate that is beyond the temporal resolution of our experiment. These excitations cannot be formed via dissociation of cold singlet states as evidenced by the PA signal not being correlated with the decay of the SE. At present, we cannot distinguish whether the formation of spatially separated excitons occurs directly during the photoexcitation or via rapid dissociation of on-chain singlet excitons during vibrational cooling. The formation of spatially separated excitons has been observed also in PPV at photon energies above the absorption threshold. In that material this process competes efficiently with the formation of luminescent excitons, indicating that the formation of electron-hole pairs (e-h) is more efficient in PPV than in LPPP. This is probably due to the bulky substituents in LPPP, which prevent short interchain separation that facilitate charge transfer. The initial fast decay of the PA occurs with a time constant of $\tau \approx 2$ ps. It can be ascribed to the collapse of a nearest-neighbor e-h pair. The deviation from exponentiality at longer times can be accounted for by a distribution of jump rates resulting from the onset of non-nearest-neighbor jumps.

V. CONCLUSION

We have performed time-resolved spectroscopy experiments on a ladder-type polymer (LPPP) within the time domain 0.5–100 ps. Three processes were observed: (i) strong stimulated emission from bulk excitons with an observed lifetime of 5 ps. For longer times the decay becomes nonexponential. Data taken by the technique of luminescence up-conversion bear out identical behavior. (ii) Aggregate states are populated via excitation transfer from bulk states. (iii) Photoinduced absorption is observed in the spectral region below the pure electronic transition. Due to the observed differences in the decay pattern of SE and PA, we attribute the PA signal to spatially separated excitons. In contrast to PPV the SE signal in LPPP can be observed at excitation energies well above the optical gap. The efficiency of producing spatially separated excitons appears to be less in LPPP so that this material is a promising candidate for optoelectronic devices because of its higher probability for singlet exciton formation.

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